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# In-situ nanoindentation of titania microspheres with different crystallinity

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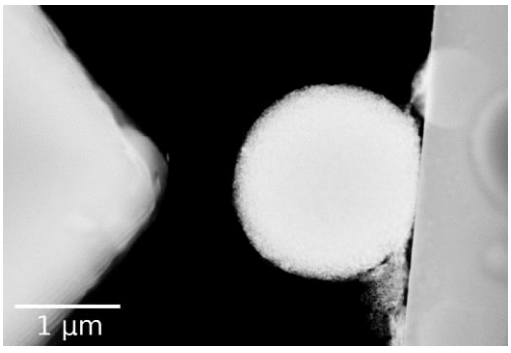
Titania microspheres are the perspective object for the fabrication of high performance liquid chromatography (HPLC) columns. The usual material for this application are silica microspheres of ⌀1-10 μm. The proposed method of $Ti(^nBuO)_4$ controlled hydrolysis allows one to obtain titania microspheres with high specific surface area (up to 300 $m^2/g$), predefined size (from 0.3 to 1.5 μm) and narrow size distribution (σ ranging from 10 to 15 %, Fig. 1a). Initially amorphous particles present an opportunity to obtain materials with dissimilar crystallinity by choosing the appropriate treatments. Following treatments were used for partial crystallization of particles: annealing at 400°C, annealing at 700°C, hydrothermal treatment (HT), hydrothermal treatment with subsequent annealing at 400°C. Mechanical properties of the individual particles from the untreated and treated samples were measured *in-situ* with the use of the MEMS-based Hysitron PI-95 at Zeiss Libra 200MC TEM (Fig. 1b). Particles were sedimented on the silicon wedge (1 μm flat-top) by dry coating. Compression tests at constant rates under displacement and load control as well as cyclic loading with partial unloading were performed. Observation of the indentation processes were performed in HAADF-STEM mode, corresponding video data were acquired.

In order to perform graphical data treatment a Python-based software was developed. For the indenter tip tracking the Digital Image Correlation (DIC) method was used. Obtained data allows us to estimate the drift function with the use of theoretical and calculated displacements, and to quantify the shape evolution of the particles.

The reduced Young's module was evaluated implying axially symmetric deformation. It was demonstrated, that partially amorphous HT-processed $TiO_2$ particles have a lesser strength limit in comparison with both the initial amorphous and annealed crystalline particles. This effect is presumably caused by the heterogeneous spatial distribution of the amorphous and crystalline phases in the HT-processed sample.

(a) 

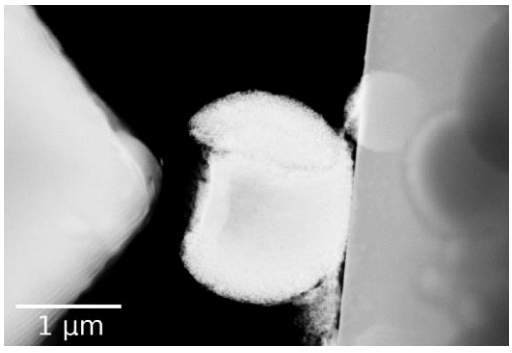
(b) 


Figure 1. Titania microspheres, STEM: (a) before the indentation, (b) after the indentation.

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